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REACTIONS AND RELAXATION OF VIBRATIONALLY EXCITED HYDROGEN

January 23, 1981

Final Report on AFOSR Contract AF F49620-79-C-0154

Jerome V. V. Kasper, Principal Investigator

An EMR vacuum UV photomultiplier tube was acquired and installed. This tube has a lithium flouride window and is solar blind. The mounting system for this tube had to be evacuable to below 50 microtorr in order to avoid catastrophic failure of the tube. The tube is constructed with an integral half-O-ring on the front around the window. This permitted us to fairly readily attach the tube to the exit window flange of the sample cell. A small tube connected to the McPherson vacuum UV monochromator provided an adequate vacuum for operation. The tube performed as expected and permitted us to obtain spectra of hydrogen in a modified experimental configuration. In this configuration, the sample cell was located between the monochromator and the detector, instead of between the source and the monochromator. A serious problem in the past was degradation of the lithium flouride cell windows due to color center formation. The magnitude of the degradation was sufficient to give us a signal loss of 5% per minute for over 4 orders of magnitude loss. Placement of the cell after the monochromator markedly reduced this degradation. In fact, after a rapid initial degradation of approximately 20-30%, the continued degradation is less than 1% per hour. This loss is principally due to color center formation in the one window re-

maining between the Argon continuum lamp and the vacuum monochromator. The reduced degradation was possible because this one window is positioned immediately adjacent to the entrance slit and is over 25 cm from the source discharge. The marked decrease in degradation rate was achieved with no loss of signal intensity. In fact, replacement of the sodium salicylate/visible photomultiplier detector with the new vacuum UV detector gave a substantial increase in signal.

Following several months of reliable operation, the vacuum in front of the tube ceased to be adequate for operation. The photocathode is located at the lithium fluoride window. According to the manufacturer, the tube will fail catastrophically if the high voltage is applied when the pressure on the window is above 50 microtorr. Over two months of leak testing, redesign, and reconstruction were required to solve this problem. Unfortunately, the tube potting material developed a slight crack that permitted air to diffuse from the unevacuated rear of the tube to the front. It was thus necessary to construct a second pumping station to evacuate a redesigned cell mount which permitted the entire cell to be contained within the vacuum.

One unexpected result of the new experimental configuration was an increase in noise due to electrical pickup. Some rewiring and shielding was found to be necessary. In particular, pickup from the lamp at times made operation almost impossible without

substantial shielding thereof. An improved electronic filter was designed, constructed, and tested. The major component of the remaining noise has its source in lamp fluctuations and is of very low frequency. Spectra obtained at slower scan speeds with increased filtering show no significant improvement in signal-to-noise. The spectrometer software was modified to permit routine acquisition and averaging of data from multiple shorter scans.

Isothermal calorimetry is used to determine the concentration of hydrogen atoms. Although we had a working, self-balancing Wheatstone bridge, the solid-state amplifier and power transistor failed due to a short in the power supply. The components were no longer available and much effort was expended in a fruitless attempt to redesign the circuit. We finally returned to a manually-balanced bridge circuit. The hydrogen atom concentration is one of the important variables which must be known for the proposed research. A series of experiments with different probes were performed to determine efficiency of atom recombination thereon. The absorption of atomic hydrogen resonance radiation was used to determine relative atom concentrations. A probe design and preparation method was found which gave the probes a better than 90% efficiency. Computer software was developed to permit the rapid and accurate calculation of atom concentration from the measured voltages, flow rates, and pressures.

Stability of atom concentration both in time and over the length of the flow region is critical. An extensive series of tests were performed to determine which coating was the best for our purposes. A coating of Teflon has proved in the past to be adequate both at preventing wall recombination of hydrogen atoms and deexcitation of vibrationally-excited molecular hydrogen. The nature of the surface depends on both the quality of the Teflon and the method of preparation. The size and shape of the new flow tubes caused the method to be different. The resulting surface proved to be inadequate. Coating of the tubes with phosphoric acid was then tried. Several different concentrations of acid were tried. Measurements on all these showed that coating with 85% acid and partial drying gave surfaces with a recombination rate for atoms of less than 1 per second. The major remaining source of instability is the microwave power supply used for production of atoms. At the lower powers required for our experiments, the supply has proved to be particularly unstable. The atom concentration nevertheless can be made stable to better than 20%.

Extensive modeling was performed to permit complete specification and design of the temperature-controlled reactor. Calculations of the diffusional mixing time under our experimental conditions were carried out to verify that our design was adequate to the task. Further calculations showed that back-diffusion from the point of mixing would not have a substan-

tial effect. Limits on flow rates that would permit the temperature to reach the desired values were determined. A double-walled variable mixing-time reactor was then designed and constructed.

Various improvements were made to the data-acquisition and display software. Software to drive the HP 7225A xy-plotter was not available and had to be written. The curve-of-growth method used for determination of the concentration of vibrationally- excited hydrogen requires good spectra with an accurately known baseline. Due to drifts in the electronics, the baseline must be determined for every spectrum taken. The slit flap valve was automated via a computer controlled stepping motor. The software was modified to close the valve prior to every spectrum and open it after approximately 20 data points were taken. When closed, no source radiation reaches the detector and an accurate baseline results. Thus the baseline is an inherent part of every spectrum and is averaged along with the spectrum. Several other hardware modifications were made. For example, the strip chart recorder was interfaced in such a way that the computer could control its operation. The control software was improved to permit graphical presentation of the averaged data as acquired. Thus data can be acquired until it is of sufficient quality; the point at which this occurs is now readily apparent.

The progress of this research was hampered by various hardware failures. The disk system on the control computer failed and was returned to the manufacturer for repair. It was returned after a month and failed within 24 hours. When it was finally repaired and the system again operational, the Baratron pressure gauge failed. Almost 6 months with much hassle were required before this pressure gauge was finally repaired in a satisfactory fashion. Although the contract period is over, work is continuing on the proposed research with limited funding from UCLA.